

REMARKS

Reconsideration of the application is respectfully requested.

Claims 1-12, 14-16, 18, and 20 are currently pending. Claims 13, 17, and 19 were previously cancelled.

Double Patenting

Claims 1-12, 14-16, 18, and 20 stand provisionally rejected over USSN 10/772,823.

Upon indication of allowable subject matter in the present case, an appropriate Terminal Disclaimer will be filed.

Rejection Under 35 U.S.C. §102 and/or 35 U.S.C. §103

The Action maintains the rejection of Claims 1-12, 14-16, 18, and 20 under 35 U.S.C. § 102(b) as being anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over U.S. Patent No. 5,124,418 to Welborn (Welborn.) Applicants respectfully disagree.

Applicants have previously clarified that in the instant bimodal polymer, the high molecular weight component has a molecular weight distribution between 4.5 and 6.88. The Action notes that Welborn discloses a molecular weight distribution range from 2.5 to 100. The broad range demonstrates that Welborn fails to disclose or suggest a polymer having the properties of an inventive polymer produced according to Applicants' presently claimed invention. In order to facilitate prosecution, Applicants provided test results to show the novel and non-obvious difference of polymers produced according to Applicants' presently claimed invention over the polymers produced according to Welborn.

On Page 11, the Action asserts that Applicants have failed to provide the notebook pages to substantiate the argued criticality of the claimed invention since Applicants failed to provide any experimental conditions for preparing the resins of Welborn.

Applicants submit the following in proof that the comparative polymer was produced according to the Welborn disclosure. The following data was presented in “EMCC BIMODAL TECHNOLOGY DISCLOSURE” presented to Univation Technologies.

Slide 36 discloses the following:

EMBC CATALYSTS

EMBC catalyst is a bimetallic catalyst containing a Ziegler component and a metallocene component.


Ti sites (Ziegler component) produce the HMW polymer component.

Zr sites (Metallocene component) produce the LMW polymer component.

The catalyst formulation needed to obtain the proper weight fraction of the HMW polymer component depends upon the activity of the Ziegler component.

Water addback is used to control the weight fraction of the HMW polymer component.

TMA (trimethylaluminum) is needed as a cocatalyst.

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Slides 37-40 disclose the process by which the catalyst (referred to in the data as EMBC) was produced.

PREPARATION OF EMBC

955 Silica $\xrightarrow{\text{calcined}}$ Calcined silica

Calcined Silica $\xrightarrow{\text{Paraffin}}$ $\xrightarrow{\text{DBM solution}^*}$ $\xrightarrow{\text{1-Butanol}}$ $\xrightarrow{\text{TiCl}_4}$ $\xrightarrow{\text{Dry}}$ CIS-2

Paraffin: Isopentane, Isohexane, Heptane

Temperature: 50-55°C

*15-17 wt% DBM (dibutylmagnesium) in heptane or hexane

MAO solution (30 wt% toluene) $\xrightarrow[\text{ambient}]{(\text{n-BuCp})_2\text{ZrCl}_2}$ MAO/Met. solution

CIS-2 $\xrightarrow{\text{Paraffin}}$ $\xrightarrow[\text{ambient}]{\text{MAO/Met. solution}}$ $\xrightarrow[45-50^\circ\text{C}]{\text{Dry}}$ EMBC

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EMBC FORMULATION

	EMBC-I	EMBC-II
Silica	1.00 kg 955-600	1.00 kg 955-800
DBM	0.72 mol	0.72 mol
1-Butanol	0.684 mol	0.74 mol
TiCl ₄	0.432 mol	0.432 mol
MAO	7.1 - 7.8 mol	6.7 - 7.8 mol
(n-BuCp) ₂ ZrCl ₂	59 - 65 mmol	67 - 78 mmol
Al (MAO)/Zr	120	100

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SILICA CALCINATION TEMPERATURE

	955-600 silica	955-800 silica
Surface hydroxyl concentration	0.7 mmol/g	0.45 mmol/g
Relative CIS-2 productivity	1.00	1.25

Ti efficiency of 955-800 based CIS-2 is significantly higher than that of the 955-600 based CIS-2.

Increasing the calcination temperature produced a more active CIS-2 component without using a higher loading of TiCl_4 .

Higher loadings of TiCl_4 may be deleterious to Zr efficiency.

A more active Ti component will require higher levels of H_2O in the process to increase the Zr efficiency and produce resins with the target weight fractions.

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Ti-BASED CIS CATALYST PRECURSORS

General scheme for preparation of CIS (Chemical Impregnation on Silica) catalyst precursor:

Calcined silica $\xrightarrow{\text{Paraffin}}$ $\xrightarrow{\text{MgR}_2}$ $\xrightarrow{\text{Modifier}}$ $\xrightarrow{\text{TiCl}_4}$ $\xrightarrow{\text{Dry}}$ CIS precursor

MgR₂: Dialkylmagnesium compound usually DBM (dibutylmagnesium)

Temperature: 50-55°C

Paraffin: Isopentane, Isohexane, Heptane

Modifiers: Acid Anhydrides, Acid Chlorides, Alcohols, Aldehydes, Alkoxysilanes, Carboxylic Acids, Chlorinated Hydrocarbons, Chlorinated Silanes, Esters, Ketones

Whole range of product applications

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Slides 56-58 disclose the process by which the polymers were prepared using the comparative "EMBC catalyst".

PROCESS DESCRIPTION

- Dry catalyst feed.
- TMA co-catalyst is used. This co-catalyst also makes EMBC relatively tolerant of reactor feed impurities.
- Ethylene feed for EMBC can contain at least 1.2 ppm O₂, 1.3 ppm CO₂, or 4 ppm acetylene with no impact on catalyst productivity or static. There is no need for special purification.
- Target product FI (split) is controlled precisely by water feed
 - Water in the presence of TMA activates Zr site
 - Does not cause process upset, although water does increase productivity
 - Keeps the HMW/LMW component properties within targets
- EMBC has made a wide range of products in the pilot plant
 - HLMI from 4 to >15
 - Density from 0.932 to 0.957
 - Recent experience at 0.945 density was favorable

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PROCESS DESCRIPTION

GRADE DIRECTIVES					
Parameter	Units	Min	Target	Max	
Production Rate	kg/h	20000	25000	30000	25000 kg/hr = 7.5 STY
Reactor Temperature	deg C	93	95	97	
Hexene/Ethylene feed ratio	kg/kg	0.01	0.013	0.016	Use this to control product density
Water feedrate	wppm on C2	15	20	35	Use this to control Flow Index
Reactor Pressure	barg	20.2	20.7	21.2	
Hydrogen/Ethylene Mole Ratio	mole/mole	0.010	0.011	0.012	
Hexene/Ethylene Mole Ratio	mole/mole	0.006	0.009	0.011	
Ethylene Partial Pressure	bar abs	10	10.85	11.5	
Isopentane Concentration	mole %	10	12	14	
Weight % Condensate	wt %	5	8	12	
Catalyst Productivity	kg/kg	3000	4500	6000	
TMA feedrate	kg soln/ton C2	0.13	0.15	0.22	0.15 kg soln/ton C2 = 75 wt ppm on PE
Cycle Gas Superficial Velocity	m/s	0.72	0.74	0.76	
Bed Level	m	13.3	13.5	13.7	
Nitrogen Flowrate to Purger	kg/h	300	450	600	
Steam Flowrate to Purger	kg/h	1	3	4	
REACTOR CONCENTRATIONS (MOLE%)			Target		
Ethylene			50.0%		for 300 psig reactor pressure
Hydrogen			0.55%		
Hexene			0.45%		
Isopentane			12.0%		
Ethane			15.0%	17%	
Nitrogen			22.0%		

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SCALE-UP: KEMCOR G-1750, April 1997

- Produced 240 tons prime feedstock in 8 days of continuous operation
 - 100 tons pipe
 - 140 tons HMW Film
- Some skins / sheeting on startup (low bed level, inadvertent high catalyst feed)
- Temperature control problems
- TMA leak to flare made control difficult
- Some rubble formation
- Plugged compressor suction screen due to poor bed level control
- No compounding was done in Australia

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Accordingly, as the data shows, EMBC is a bimetallic catalyst containing a Ziegler component and a metallocene component according to the Welborn disclosure. Furthermore, the polymers produced, which are comparative examples in the instant disclosure, were produced according to the Welborn disclosure.

Previously submitted Attachments 4 and 5 are excerpts from the report entitled “DRAFT Review of Maxis PE100 Performance” Dated February 2000 (“the Maxis Report.”) The comparative material for purposes herein is referred to in the Maxis Report as HDX891. The following excerpt is provided from the Maxis Report showing that the comparative material referred to in Slide 58 herein as KEMCOR G-1750, April 1997 is the same comparative polymer referred to in the Maxis Report as HDX891.

2 Summary

The potential of Maxis to make a PE100 grade was developed using pilot plant samples. These samples were used to develop a performance-based specification. In May '97 a number of commercial blends of Maxis were made to this specification. These blends were tested to the pipe performance standard. There are three critical pipe performance requirements. They are:

- . Hoop stress life as a function of temperature,
- . Resistance to slow crack growth, - SCG
- . Resistance to rapid crack propagation. - RCP

The May '97 blends, HDX891, failed the last two of these three critical performance requirements.

Hoop stress lifetime testing on HDX891 was terminated before the full analysis had been completed. Extensive testing of pilot plant samples has shown that these materials are likely to meet the hoop stress lifetime requirement without a knee being detected in the 80 °C data. However there maybe a knee in the 80°C hoop stress curve of the HDX891. The data available indicates that HDX.891, while potentially meeting the standards hoop stress lifetime requirements, would not be a second generation PE100.

There appears to be some scope to improve the SCG resistance of Maxis, however improving the RCP performance of the polymer will require an improved understanding of the effect of molecular structure and compounding variables on RCP.

The HDX891 manufactured during the May '97 trial was at the low end of the weight fraction hmw specification. This could have adversely affected its performance.

Note – there are substantial differences between the US and European/Australian pipe compound standards. This report compares Maxis grade HDX891 to the requirements of the European/Australian standards for pipe compounds.

4.1 Background

4.1.1 Compounds tested

Maxis pilot plant samples were tested as natural material. These samples were compounded on a single screw extruder together with primary and secondary antioxidant package. The May '97 plant trial samples (HDX891) and all controls were fully formulated pipe compounds. Full formulated pipe compounds contain 2.25% carbon black and for the Maxis HDX891 grade the associated LD master batch carrier.

4.2 Product Analysis

Two blends of HDX 891 were produced during the May '97 commercialization trial, blend 3714 and 3706. These blends were produced on the Qenos Plastics Banbury using commercial compounding conditions and a LD based master batch. Table 4.1.1 contains the property data for the materials produced during the trial.

Sample HDX891 blend 3714 was chosen for extensive testing as it was produced later in the trial, and the properties were closer to where we believed we would like to operate in the longer term.

Applicants note that the Kemcor G-1750 material produced in April, 1997 was subsequently compounded as a pipe resin as disclosed in Section 4.2 Product Analysis above. This resulted in the HDX891 blend 3714 which serves as the comparative data according to the Welborn disclosure for purposes herein.

Data Tables 4.5.1 and 4.5.2 provide Rapid Crack Propagation testing according to ISO-13477. Applicants note, the minimum requirements of the testing are >7 bar. If a material is unable to meet these minimum requirements, the Predicted Tc according to the testing is not conducted. As the data in Tables 4.5.1 and 4.5.2 show, the comparative polymer of Welborn did not meet the minimum pressure requirements according to ISO-13477, thus the Tc was not determined. However, as is shown in Attachment 1, the inventive polymer greatly exceeds the minimum requirements of ISO-13477 and thus a Tc was determined. This limitation is currently recited in the presently claimed invention.

4.5 Rapid Crack Resistance Analysis

Resistance to the propagation of **high-speed crack** is a critical performance criterion for PE100 grades. Rapid crack propagation (RCP) resistance is measured in either a full-scale test or the small scale S4 test. The full-scale test uses 50 m specimens of 500mm OD pipe. It is an expensive test requiring large quantities of material. The S4 RCP test correlates to the full-scale test. It uses two meter lengths of 250mm OD pipe. Ten to fifteen specimens are required for the S4 evaluation. RCP tests are appropriate for commercially produced materials but not useful for prototype development. The Charpy impact test was used to evaluate Maxis PE 100 prototypes.

The S4 RCP test was carried out on Maxis HDX891 blend 3714. It did not meet the RCP requirements of a PE 100 material.

4.5.1 S4 RCP evaluation – ISO 13479

Samples of HDX891 blends 3706 and 3714 were extruded into 250mm OD pipe. Pipe from blend 3714 was tested, to ISO 13479 the S4 RCP test, at South West Research Institute, USA. The S4 test determines the critical internal pressure at which the pipe fails in an unstable fashion.

4.5.2 Charpy Impact testing.

The Charpy impact resistance of the polymers was determined using two impact techniques. Both tests determine the critical energy for crack initiation G_c . Test one used large specimens 10x30 mm cross section and an instrumented impact tester. Test two used a conventional Charpy specimen with a sharp notch and a conventional pendulum impact tester. Information from a commercial supplier of PE100 grades suggested that using test method two, a G_c greater than 12 kJ/m² is required to meet the ISO RCP requirements. Data from these two tests is given in Tables 4.5.1, 4.5.2 and 4.5.3

Table 4.5.1 Charpy and RCP results – Test method one

Sample	Charpy Impact, 0 °C, (kJ/m ²)	S4 RCP Test, ISO – 13479 (bar)
AS 4131 minimum requirements	N/A	>7
PE100 Control (CRP100)	38	>25
PE100 (LBH1133)	42	>17
Commercial PESO Grades		
MD0898	5	1.25
HD2468	6	0.5
May '97 Product		
HDX891 -- 3706	7	
HDX891 – 3714	4	1.0
HYA 020	11	1.4
Pilot Plant Prototype Samples		
160303D	17	-
260720B	6	
260815C	18	
260815D	18	

Table 4.5.2 Charpy and RCP results – Test method two. Target $G_c > 12 \text{ kJ/m}^2$

Sample	Charpy Impact, 0 °C, (kJ/m ²)	S4 RCP Test, ISO – 13479 (bar)
AS 4131 minimum requirements	N/A	>7
PEIOO Control (CRP100)	11.6	>25
PEIOO (LBH1133)	11.0	>17
Commercial PESO Grades		
MD0898	4.8	1.25
May '97 Product		
HDX891 – 3714	5.1	1.0
HYA 020		1.4
Pilot Plant Prototype Samples		
260720B	6.7	
260815C	9.8	
260815D	9.0	-
160226C	11.5	-

Table 4.5.2 shows that HDX891 from May '97 passed neither the G_c or RCP criteria.

The data previously submitted in Attachments 1, 2, 3, 4, and 5 are summarized in the following table as they pertain to the differences in the Comparative material of Welborn as compared to Applicants' presently claimed invention.

	Comparative Polymer of Welborn	Source of Data	Inventive Polymer	Source of Data	Comments
ISO-13479 Slow Crack Growth (hours) 5 MPa hoop stress, 80°C	107 hrs	Attch. 5	3,672 hrs	Attch. 1	Attach. 3 ISO-13479 Requirements ≥ 500 hrs.
ASTM-1473 PENT Slow Crack Growth 2.4 MPa 80°C	95 (poor)	Attch. 5			The ISO-13479 testing is more rigorous than the ASTM-1473 testing. As such, the Inventive polymer will have excellent properties based on the ISO-13479 testing.
Rapid Crack Propagation ISO-13477 (S4 RCP Test) (bar)	1 bar	Table 4.5.1 and 4.5.2	Pc > 10 bar Tc = -13°C	Attch 1	ISO-13477 testing Requirements Pc > 7

As the data shows, Applicants' presently claimed invention represents a vast improvement in physical properties over the polymer disclosed by Welborn. In fact, the polymer produced according to Welborn does not meet the minimum requirements for polyethylene tested in the form of pipe under ISO-13477 or ISO-13479. However, the presently claimed invention greatly exceeds these minimum requirements.

The values obtained in the comparative examples thus demonstrate that polymers produced according to Welborn do not inherently possess the properties obtained by Applicants' presently claimed invention. In any event, polymers produced according to Welborn fail to meet the standards associated with PE-100 grade material, which is in contrast to Applicants' presently claimed invention. Accordingly, the polymer produced

according to the presently claimed invention is both novel and non-obvious over Welborn as demonstrated by the superiority of the instant polymers over polymers produced according to Welborn.

Applicants respectfully request that all rejections be withdrawn and solicit a prompt notice of allowability. In the alternative, Applicants invite the Office to telephone the undersigned attorney if there are any other issues outstanding which have not been presented to the Office's satisfaction.

Respectfully submitted,

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Date

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